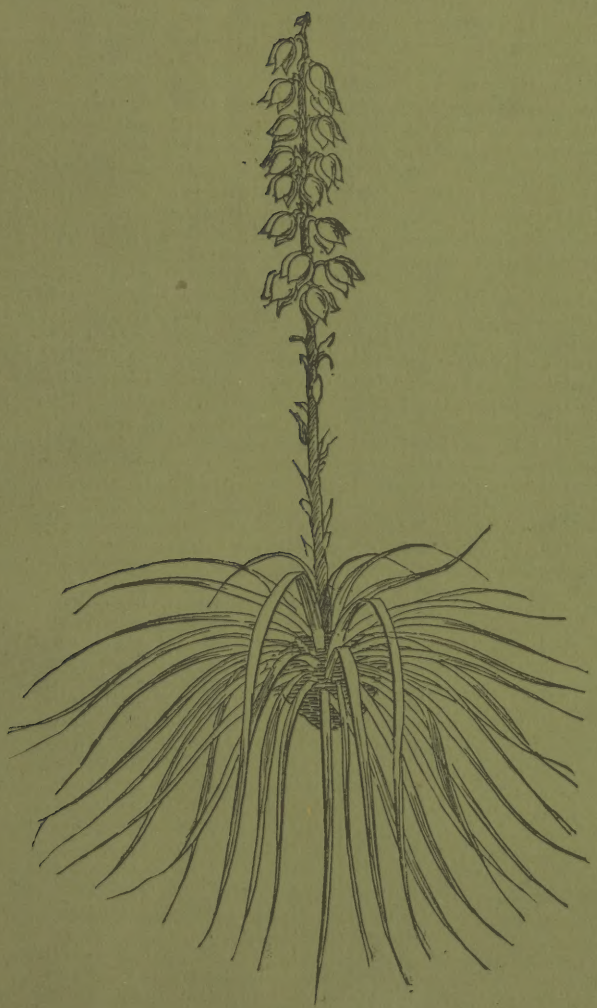
de S. Abbott. (Helen G)

Yucca

Angustifolia.

Abbott



Compliments of the Author.

YUCCA ANGUSTIFOLIA:

A CHEMICAL STUDY

BY

HELEN C. DE S. ABBOTT,

Fellow of the American Association for the Advancement of Science; Member of the Academy of Natural Sciences, Philadelphia; of the Berliner Chemische Gesellschaft, etc., etc.

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A CHEMICAL STUDY

OF

YUCCA ANGUSTIFOLIA.

BY

H. C. DE S. ABBOTT.

Read before the American Philosophical Society, December 18, 1885.

ARTICLE III.

A CHEMICAL STUDY OF YUCCA ANGUSTIFOLIA.*

BY H. C. DE S. ABBOTT.

Read before the American Philosophical Society, December 18, 1885.

This plant is well known in the West as the "soap-weed." It grows very abundantly in most of the Western States and Territories. It has attracted the attention of botanists, and it is a plant of interest on account of the many uses to which it has been put in the countries where it is found.

The results noted in this paper are based upon a first and introductory chemical analysis of the *Yucca*. Previously, little has been studied of its chemistry. It is briefly mentioned in the work of a French writer, Dr. Georges Pennetier;† also, in a paper on the study of manganese found in the ash of plants in which M. Maumené states that the ash of the *Yucca* contains manganese.‡ He does not name what species of *Yucca* was examined. The former writer gives the micro-chemical characters of the action of iodine and sulphuric acid, dilute chromic acid, and cuprammonia on the fibres of the *Yucca angustifolia*.

The specimens of *Yucca* used in these analyses were of large growth and in good condition. The entire plant was examined, and a separate study made of the bark and wood of the root, and of the green leaf and the yellow basal part. The roots were air-dried, freed from adherent dust, reduced to a very fine powder, and passed through a No. 80 sieve. The leaves were less finely powdered.

Dragendorff's scheme for plant analysis§ has been generally followed. Ten grams of the air-dried powder were used for the preliminary examination of soluble substances. For every gram of the powder, ten c. c. of the solvents were employed. An additional quantity of the powder was prepared for special purposes. Five grams of the air-dried powder were dried, in a hot-air oven, at a temperature

* An abstract of this paper was read before the Chemical Section of the American Association for the Advancement of Science, at Ann Arbor, Michigan, August 28, 1885.

† Leçons sur les Matières Premières Organiques, Paris, 1881, p. 446.

‡ M. E. J. Maumené, Bul. de la Société Chimique de Paris. Tome xlii, p. 305.

§ Plant Analysis. Qualitative and Quantitative, by G. Dragendorff. Translated from the German by Henry Greenish, London, 1884.

between 100° C. and 110° C. until the weight remained constant, for the estimation of moisture. This powder was incinerated in a covered porcelain crucible at a dull red heat until the carbon was entirely consumed. The per cent. of total ash was determined from it.

QUALITATIVE ASH ANALYSES.

Calcium, magnesium, potassium, sodium, iron, manganese, chlorides, phosphates, and sulphates, were found in every part of the plant.

Determination of { I. MOISTURE / II. TOTAL ASH } on the powder.

	I.	*II.*	*Color of Ash.*
(1.) The bark of the root.....	6.78 per cent.	17.38 per cent.	reddish
(2.) " wood "	11.67 "	15.75 "	" gray
(3.) " green leaf.....	8.11 "	5.75 "	gray
(4.) " yellow base of leaf.....	37.00 "	10.63 "	white

PETROLEUM SPIRIT EXTRACTS.

Extract (1) Bark of Root.

The maceration was conducted in an apparatus similar to one described in Dragendorff's "Plant Analysis."* A light petroleum spirit was used which boiled between 25° C. and 45° C. The *extract* was filtered from the powder-residue. It was a clear pale yellow-colored liquid, and slightly acid in reaction. A drop of the *extract* on evaporating left a uniform spot on blue paper. The *extract* was evaporated at the ordinary temperature. The *residue* was a solid, and it had the odor and characteristic crystalline structure of fatty acids, suggesting the presence of a fixed oil. Its melting point was taken. The substance melted at 60° C., and on cooling solidified amorphous. To determine the total amount of solids extracted, a definite volume of the *extract* was evaporated, dried, and weighed.

TOTAL SOLIDS.

Petroleum spirit residue dried at 100° C.....	1.24 per cent of solids.
" " " 110° C.....	1.20 " " "
	0.04 " " loss.

The *residue* was identified as a fixed oil. It was soluble in petroleum spirit, ether, benzole, chloroform, amyl alcohol, carbon di-sulphide, and cold aqueous alkalies; incompletely soluble in cold or boiling 86 per cent. alcohol, 95 per cent. alcohol, absolute alcohol, acetic ether, and ammonium hydrate. No change of color was

* Page 99, Tollen's apparatus.

observed on treating the fixed oil with concentrated sulphuric acid, nor on the addition of syrupy phosphoric acid, though it was partially soluble in these acids. Phosphoric acid colored it yellow; it was colored yellowish by concentrated hydrochloric acid and nitric acid of 1.22 specific gravity. A mixture of concentrated sulphuric acid and nitric acid of 1.22 specific gravity changed the color of the fixed oil to a reddish-brown; it was colored pale green by sulphuric acid of 1.634 sp. gr. and of 1.53 sp. gr. Calcium di-sulphide gave a bright green color reaction with the fixed oil, but did not form an emulsion with it; aqueous solutions of gold and platinum chlorides were reduced by it. The fixed oil was saponified with difficulty by alcoholic soda; but readily by boiling aqueous soda; a white fragile soap was separated and filtered from the liquid. The soap was decomposed by hydrochloric acid and the fatty acids separated. The filtrate from the soap was examined for glycerin. By the method* used, an oily liquid was obtained, it was heated with anhydrous borax on platinum foil, and gave the usual green-colored flame test for glycerin. The alcoholic solution of the *petroleum spirit residue* was fractionally precipitated with an alcoholic solution of magnesium acetate, and traces of an amorphous residue were recovered.†

The *petroleum spirit residue* was digested with water containing sulphuric acid, and examined for alkaloids which are sometimes brought down with fixed oils. The usual reagents failed to detect traces of alkaloids.

Extract (2), *Wood of the Root.*

The maceration was carried out under the same conditions as in *extract* (1).

The *extract* was a clear, colorless solution, neutral in reaction. A drop of the liquid left no uniform spot on blue paper. The *extract* was evaporated at the ordinary temperature. The *residue* was light yellow-colored, of a semi-solid consistency and melted at 36° C. A definite volume of the *extract* was evaporated, dried, and weighed.

TOTAL SOLIDS.

Petroleum spirit residue dried at 100° C	0.55 per cent of solids.
" " " 110° C	0.35 " " "
	0.20 " " loss.

The *residue* was identified as a fixed oil; associated with volatile fatty acids. The latter were indicated by the 0.2 per cent of loss, and the disagreeable odor of the *residue* which was dissipated on heating at 110° C.

The *petroleum spirit residue* from the *extract* was evaporated at the ordinary temperature, dissolved with difficulty in cold 95 per cent. alcohol, and in boiling weaker

* "Plant Analysis." G. Draggendorff, p. 12.

† Loc. cit., page 16.

alcohol; absolute alcohol hardened and discolored it. Concentrated sulphuric acid, nitric acid, and hydrochloric acid, did not appreciably act on the *residue.* It was not saponified, but slowly dissolved by boiling aqueous and alcoholic soda. The alcoholic solution of the *petroleum spirit residue* was submitted to a fractional precipitation with an alcoholic solution of magnesium acetate. The first precipitation obtained was purified by boiling alcohol; it was an opaque scaly crystalline solid which melted at 85° C. The second precipitation yielded traces of a white amorphous substance. The third precipitation resulted from adding strong ammonia water to the magnesium acetate solution, and the purified residue melted at 60° C.

Negative tests for alkaloids followed an examination of the aqueous treatment of the *petroleum spirit residue.*

Extract (3), *Green Part of the Leaf.*

The method of extraction was the same as that used in the previous extractions. The *extract* was clear, pale green in color, and non-fluorescent. It was colored by a small quantity of chlorophyll, which the petroleum spirit dissolved. The liquid was acid in reaction. A drop of it left a permanent stain on blue paper when evaporating. The *extract* was evaporated at the ordinary temperature, and the *residue* was a dark greenish-yellow semi-fluid substance. The solidifying point was taken. It was found to be about 15° C. A definite volume of the *petroleum spirit extract* was evaporated, dried, and weighed.

TOTAL SOLIDS.

Petroleum spirit residue dried at 100° C	2.20 per cent of solids.
" " " 110° C	2.01 " " "
	0.19 " " loss.

The *petroleum spirit residue* was identified as a fixed oil with a small amount of chlorophyll that had been brought into solution by it. It was soluble in cold 83 per cent. alcohol, 95 per cent. alcohol, absolute alcohol, amyl alcohol, ether, acetic ether, chloroform, benzole, carbon di-sulphide and glycerin. It was also soluble in oil of turpentine, almond oil, ammonium hydrate, mercuric chloride, and slowly soluble in acetic acid. Concentrated nitric acid, and hydrochloric acid slowly dissolved the fixed oil, the former colored it dark green, and on stirring the mixture the color was changed to a brown. Concentrated sulphuric acid dissolved and changed it to a very dark-brown color; on adding concentrated nitric acid, the liquid was changed to a reddish-brown color.

The following reactions were noted: The fixed oil changed to a hard greenish-yellow substance on heating it with anhydrous borax on platinum foil. When rubbed

on a crucible lid with powdered rosaniline, it was colored red, showing the presence of free fatty acids. It did not emulsify with calcium di-sulphide nor with syrupy antimony chloride, but it was colored dark-green by the latter. It was imperfectly dissolved by phosphoric acid, and slowly soluble in equal parts of cane sugar and concentrated hydrochloric acid; more rapidly soluble in equal parts of cane sugar and nitric acid. An aqueous solution of picric acid made alkaline by sodium carbonate colored the fixed oil a light reddish-brown color, cane sugar added to the solution facilitated dissolving it. It was instantly dissolved by equal parts of picric acid and acid ammonium phosphate, and on warming with stannous chloride, leaving a turbid yellow-colored liquid. It was insoluble in aqueous barium hydrate; soluble in alcoholic ammonia with no coloration, and in sulphurous acid. It was colored brown when mixed with sulphuric acid of 1.634 specific gravity, and incompletely dissolved; it was also colored brown by ferric chloride. On adding to the fixed oil sulphuric acid of 1.475 specific gravity, and a small quantity of zinc, hydrogen was generated, and the solubility of the oil in the acid liquid was accompanied by a rosy tint given to the solution.

Extract (4), *Yellow Part of the Leaf.*

This *extract* was obtained by a similar process as that used for the other petroleum spirit *extracts.* The *extract* was a pale yellow-colored liquid. The reaction was slightly acid. A uniform spot was left on blue paper as the drop evaporated. The petroleum spirit was evaporated at the ordinary temperature, and a yellow-colored *residue* recovered, of a semi-solid consistency and crystalline in structure. It solidified at 12° C. From a definite volume of the *petroleum spirit extract*, the amount of total solids was determined.

TOTAL SOLIDS.

Petroleum spirit residue dried at 100° C.	1.1 per cent of solids.
" " " 110° C.	1.1 " " "
	0.00 " " loss.

The *residue* was identified as a fixed oil. It was soluble in warm absolute alcohol, incompletely soluble in weaker alcohol; soluble in cold acetic ether, chloroform, benzole, amyl alcohol, ether, carbon di-sulphide, and glycerin. It was saponified with aqueous soda and a white soap separated. No reaction was observed with picric acid and ammonium phosphate, nor with nitric acid of 1.32 specific gravity and 1.18 specific gravity. The fixed oil was soluble in potassio-mercuric iodide solution; and colored dark-brown by alcoholic ammonia. A mixture of ferric chloride solution and powdered rosaniline gave a fine violet-colored reaction with the fixed oil.

An examination of the aqueous treatment of the *petroleum spirit residues* (3) and

(4), for alkaloids, gave negative results. A portion of the original powder, from each of the four parts of the plant, was mixed with an aqueous solution of caustic soda, and the distillate examined for volatile alkaloids with negative results.

SUMMARY I.

Petroleum Spirit Extracts.

	Solids extracted.	*Character of residue*	*Reaction with litmus.*	*Melting point.*	*Solidifying point.*
1. The bark of the root.. ..	1.24 per cent.	fixed oil	slightly acid	60° C.	solid at ordinary temperature
2. " wood "	0.55 "	" "	neutral	36° C.	
3. " green leaf.....	2.20 "	" " chlorophyll	acid	semi-fluid at ordinary temperature	15° C.
4. " yellow base of leaf..	1.10 "	fixed oil	faintly acid		12° C.

The solids extracted by petroleum spirit from the four parts of the plant are identified as fixed oils*; associated with a volatile principle (0.2 per cent) in *extract* (2), and with traces of chlorophyll in *extract* (3).

Fixed oil (1) was crystalline in structure. It was soluble in ether, chloroform, benzole, carbon di-sulphide, and amyl alcohol; incompletely soluble in cold or boiling alcohol, acetic ether, and ammonium hydrate. It was colored pale green by sulphuric acid of 1.634 specific gravity, and changed to a bright-green color by calcium di-sulphide, but formed no emulsion with it. Phosphoric acid colored it yellow. The fixed oil was saponified, and a white soap separated. This was decomposed, and the fatty acids recovered. Glycerin was separated from the soap filtrate.

Fixed oil (2) was dissolved with difficulty in boiling 95 per cent. alcohol, and hardened and discolored by absolute alcohol. It was not saponified. Crystalline solids were separated by precipitating the alcoholic solution with magnesium acetate. They melted at 85° C., and at 60° C., respectively.

Fixed oil (3) was soluble in alcohol, ether, chloroform, benzole, carbon di-sulphide, oil of turpentine, almond oil, glycerin, and slowly soluble in acetic ether. The presence of free fatty acids was demonstrated. The fixed oil was colored dark-green by syrupy antimony chloride; on adding to it sulphuric acid of 1.475 specific gravity, and a small quantity of zinc, hydrogen was generated, and the solubility of the oil in the acid liquid was accompanied by a rosy tint given to the solution.

Fixed oil (4) was crystalline in structure. It was soluble in warm absolute alcohol, in cold acetic ether, chloroform, benzole, amyl alcohol, ether, carbon di-sulphide, and glycerin. It was saponified, and a white soap separated. The fixed oil was colored dark-brown by alcoholic ammonia, and a mixture of ferric chloride solution and powdered rosaniline gave a violet-colored reaction with it.

* Fixed Oils. Science, Sept. 11, 1885.

These fixed oils differed in their physical characters and chemical reactions. This difference may be due to the presence of free fatty acids and glycerides in varying proportions in the four parts of the plant. It is of interest to note that in the subterranean part of the *Yucca*, the oil extracted from the bark was solid at the ordinary temperature; from the wood it was of a less solid consistency; while the yellow base of the leaf contained an oil quite soft, and in the green leaf the oil was almost fluid.

Extract (2) contained an oil of low melting point. It melted at 36° C. An alcoholic solution was fractionally precipitated with magnesium acetate, and three members of the fatty acid series were isolated. The quantities obtained were small, and it was impossible to do more than to take the melting point of two of the purified crystalline residues. They melted at 85° C., and at 60° C. respectively. It is a well-known fact that a mixture of fat acids in certain proportions has a lower melting point than those of its constituents.

Alkaloids and volatile-alkaloids were not detected in the *petroleum spirit extracts.*

ETHER EXTRACTS.

Extract (1), *Bark of the Root.*

The residual powder from the petroleum spirit extraction was dried until thoroughly freed from petroleum spirit. It was then macerated with Squibbs' stronger ether in the apparatus already described. The *ethereal extract* was filtered from the powder. It was a clear crimson-colored liquid, tinted by some red coloring matter dissolved; and acid in reaction. The *extract* was slowly evaporated at the ordinary temperature; white needle-shaped crystals were seen as the liquid concentrated. The *ethereal residue* was of a resinous character. It was ruby-colored, transparent, and of a softer consistency than ordinary resin. Microscopically, the *residue* was identified as a resin by its color reaction with Hanstein's aniline violet solution.* The *ethereal residue* was treated with petroleum spirit to remove any traces of fat that may have been extracted with it. It was heated in a small tube, at 50° C. it experienced a slight change, and melted at 70° C. For a determination of the total solids, a definite volume of the *ethereal extract* was evaporated, dried, and weighed.

TOTAL SOLIDS.

Ethereal residue dried at 100° C.	3.16	per cent of solids.
" " " 110° C.	3.16	" " "
	0.00	" " loss.

The resin was incompletely soluble in 95 per cent. alcohol, absolute alcohol, and

*Botanical Micro-Chemistry. Poulsen-Trelease. Boston, 1884, p. 59.

amyl alcohol; readily soluble in ether; not appreciably soluble in chloroform, benzole, and carbon di-sulphide. It was dissolved by sulphuric acid to a colorless solution, which, on warming, turned to a yellow color, and gradually darkened to a dull brown color, fading to a pale yellow.

An attempt was made to separate the white needle-shaped crystals mentioned above. The *ethereal residue* was agitated with acetic ether. The liquid was filtered from the insoluble matter and evaporated. Traces of a resinous substance were separated. The insoluble matter was treated with boiling ether, filtered hot, and the filtrate concentrated. On cooling, the white needle-shaped crystals reappeared. They were insoluble in water and in acetic ether.

A separate portion of the *ethereal extract* was evaporated, and treated with warm distilled water. The aqueous extract was made up to a definite volume, and a known quantity evaporated, dried, and weighed. The amount of total solids was almost inappreciable by weight. The aqueous extract was not colored by iron salts, and it did not form a precipitate with alum and gelatine solution, lead acetate, potassio-mercuric iodide, nor gold chloride solutions; showing absence of tannin, gallic acid, and alkaloids. The *ethereal extract* was directly tested for these compounds, and with negative results. A portion of the aqueous extract was evaporated to dryness, and treated with potassa solution, and the residue dissolved with no coloration. Another portion of the aqueous extract was agitated with acetic-ether, and the liquids were separated; on evaporating the acetic ether solution, traces of a residue were obtained which sulphuric acid acted upon. A resinous substance separated from the greenish-colored acid liquid, the former was partially disintegrated by cold water.

The specific gravity of the resin was 1.091.

Extract (2), *the Wood of the Root.*

The residual powder from the petroleum spirit extraction was macerated in stronger ether. The *ethereal extract* was of a reddish-yellow color; slightly acid in reaction. It was slowly evaporated at the ordinary temperature, and as the liquid concentrated, white needle-shaped crystals appeared, and presented the same physical structure as the crystals found in the *ether extract* (1).

The *ethereal residue* was identified as a resin. It was a transparent, ruby-colored substance, and acid in reaction. It was heated to 50° C., at that temperature its color deepened, and at 70° C. it melted. The specific gravity of the resin was 1.091. A definite volume of the *ether extract* was evaporated, dried, and weighed to determine the amount of total solids.

TOTAL SOLIDS.

Ethereal residue dried at 100° C.	1.70	per cent. of solids
" " " 110° C.	1.45	" " "
	0.25	" " loss.

The resin was examined by Hirschsohn's scheme* with a view to classify it with known resins. It was imperfectly soluble in 95 per cent. alcohol and chloroform; soluble in ether. The alcoholic solution gave a turbidity with lead acetate, not cleared upon boiling, and with ferric chloride formed a clear mixture. Concentrated sulphuric acid dissolved the resin, leaving a dark yellow-brown liquid which faded to a dull yellow color. The sulphuric acid solution when mixed with alcohol, changed to a pale gray color. On addition of water to the acid solution, there was no coloration nor separation of the resin. Alcohol containing hydrochloric acid gave no color reaction with the resin. Bromine solution added to the chloroform-resin extract, and iodine solution to the ether-petroleum-resin extract, gave no reactions. Sodium carbonate at the ordinary temperature had no effect on the resin, but on boiling the liquid was colored yellow.

By the above examination, this resin was thrown out of the numerous classes of described resins. It is proposed to name it yuccal.†

Yuccal was soluble in boiling absolute alcohol and acetic ether; incompletely soluble in benzole, carbon di-sulphide, alcoholic ammonia, and cold acetic ether. The red color of the resin was removed by cold acetic ether, a transparent substance remaining, soluble in hot acetic ether.‡ Yuccal was dissolved by potassio-mercuric iodide. It reduced aqueous solutions of gold and platinum chlorides. A blood-red color reaction was obtained by warming a small quantity of the resin on a crucible lid with a crystal of ammonium molybdate and a few drops of nitric acid. On adding to the resin mixture a few drops of strong sulphuric acid, and again warming, it was dissolved. Warm dilute nitric acid dissolved the resin, colorless; cold nitric acid gave a brownish-green color reaction.§ Yuccal was heated on platinum foil, and as it decom-

* E. Hirschsohn. Watts Chem. Dict. Vol. viii, Pt. ii, p. 1743.

† I suggest that in future all resins be distinguished by the terminal syllable *al*, for uniformity of resin nomenclature. Yuccal; Science, September 11, 1885, p. 210.

‡ I have examined the action of acetic ether as a solvent for resins. Cold acetic ether dissolved ordinary resin, turpentine, styrax, tolu-balsam, mastic, elemi, Canada-balsam, Peru-balsam, copaiba-balsam, Venice-turpentine, and incompletely spruce-gum, and yuccal. In hot acetic ether spruce-gum and yuccal were soluble. The following resins were insoluble in hot or cold acetic ether, guiacum, sandarac, shellac, benzoin, olibanum, ammoniac, myrrh, galbanum, and asafœtida.

§ A reddish-yellow decomposition product resulted from the action of nitric acid on many resins which followed generally quite soon after adding the acid to a small quantity of the resin (0.1 gram of the resin and 5 c. c. of nitric acid 1.4 sp. gr.). But the reaction which took place varied according to the conditions, *i. e.*, strength of acid used,

posed the fumes that were given off were pleasant and aromatic. Tests failed to show the presence of benzoic or cinnamic acids.

The *ethereal residue* was treated with warm water, and on cooling, the liquid was agitated with acetic ether, which was separated, and when evaporated yielded a small quantity of resinous substance. The *ethereal residue* insoluble in water was treated with boiling ether, and as the liquid concentrated, the white needle-shaped crystals were seen floating in it, but on further concentration they could not be seen, and a yellow greasy-looking mass settled in the bottom of the beaker. On driving off the ether, a transparent and ruby-colored resinous substance remained. The aqueous extract obtained in the way described above gave no coloration with iron salts, and no precipitate with gelatine and alum solution, potassio-mercuric iodide, or gold chloride solutions. Fehling's solution was not reduced by boiling, though the aqueous extract was boiled with acid, then rendered alkaline before adding the copper test. The preceding tests gave negative results for gallic acid, tannin, alkaloids, and glucosides. A portion of the aqueous extract was acidified and agitated successively with different solvents, for glucosides, bitter principles, and alkaloids which may be removed from solution by this means. The acid liquid was then rendered alkaline with ammonia, and agitated successively with the same order of solvents that were used with the acidified liquid. No solids were separated by these methods. The *ethereal residue* insoluble in water was treated with alcohol; and yielded traces of a resinous substance. The residue insoluble in water and alcohol was not dissolved by ether, acids or alkalies.

Yuccal or the *ethereal residue* soluble in ether and alcohol was saponified, and the soap boiled with lead acetate. The yellow masses were collected on a filter and treated with boiling ether, and the filtrate was slowly evaporated. The residue was a granular solid. This substance was imperfectly purified by repeated boiling with ether, and a solid of crystalline structure obtained. It gave an acid reaction with litmus, and a red color with concentrated sulphuric acid. The acid dissolved a substance enclosing the crystals, leaving the structure of the latter uninjured and colored. Strong nitric acid dissolved the crystals with no coloration. They were soluble in absolute alcohol, amyl alcohol, benzole, chloroform, glycerin, and a solution of alcoholic soda; soluble in potassium iodide, potassium chromate, mercurous nitrate, cobalt nitrate, potassium ferro-and-ferri-cyanide solutions; insoluble in ammonia and aqueous alkalies.

the application of heat to the resin acid mixture, or the addition of solvents to the mixture. The more concentrated the acid the more rapid was the reaction. The application of heat also hastened the change, especially if a more dilute acid was used in the mixture. Some solvents acted like heat by increasing the energy of the reactions. Alcohol and ether were active solvents, and the reaction was attended by the escape of nitrous fumes from the combination of alcohol or ether and nitric acid. Chloroform and benzole were indifferent. Amyl alcohol acted feebly.

Yuccal was treated with spirit of different strengths, as a means of separating resin acids if any were present. It was treated with 85 per cent. spirit; an opaque brown substance was left undissolved, which was soluble in absolute alcohol; insoluble in ether, and colored brown by concentrated sulphuric acid. The color was not discharged by alcohol or ether. The 85 per cent. spirit solution was evaporated, and the residue treated with 50 per cent. spirit and a small quantity of a brown residue was insoluble. The 50 per cent. spirit solution on evaporating left a non-crystalline, transparent, reddish-colored solid; acid to litmus. It was colored cherry-red by concentrated sulphuric acid, and slowly dissolved to a yellowish-red liquid.

Extract (3), *the Green Part of the Leaf.*

The residual powder from the petroleum spirit maceration was thoroughly dried, and again placed in the percolator. It was treated with Squibbs' stronger ether. The *extract* was a deep-green colored liquid and fluorescent. The reaction was slightly acid. Alcohol, benzole, and petroleum spirit added to the *ethereal extract* did not cause a precipitation. An amorphous and green-colored residue was obtained on evaporating the *extract*. The amount of total solids was estimated from a definite volume of the *extract*, which was evaporated, dried, and weighed.

TOTAL SOLIDS.

Ethereal residue dried at 100° C.	1.25	per cent of solids.
" " " 110° C.	1.14	" " "
	0.11	" " loss.

The *ethereal residue* was brought into a state of fine division and treated with water. The amount of total solids soluble in ether and water was 0.34 per cent. The aqueous extract was neutral in reaction. It was faintly colored and slightly bitter to the taste. It was not colored by iron salts nor precipitated with alum and gelatine solution, showing absence of gallic acid or tannin. Copper solutions were not reduced, indicating absence of glucosides, though the precaution was observed of boiling the aqueous extract with acid and rendering alkaline before adding the copper solution. The aqueous extract was agitated with acetic ether and a distinctly crystalline residue separated. Under the microscope these crystals were white, needle-shaped and arranged in bundles. They did not respond to tests for gallic acid. Potash solution formed a yellow mixture with the crystals. The color was discharged by a drop of hydrochloric acid. Chloroform did not dissolve any substance from the *ethereal residue*. The *ethereal residue* was treated with acidulated water and tested negatively for alkaloids.

The *ethereal residue* insoluble in water was treated with alcohol. The amount of

substances insoluble in water, and soluble in ether and alcohol was 0.15 per cent. The alcoholic solution was evaporated, and the residue was crystalline in structure. Concentrated sulphuric acid imperfectly dissolved it, and gave a reddish-yellow color reaction; acetic ether discolored the solution. The alcoholic residue was insoluble in acetic ether, cold and boiling aqueous alkalies; soluble in chloroform. It saponified with alcoholic soda.

The amount of the *ethereal residue* insoluble in water and alcohol was 0.65 per cent. It was not soluble in alcoholic or aqueous soda. This would indicate a resin anhydride. Concentrated sulphuric acid gave no color reaction with it; and a mixture of sulphuric acid and cane sugar dissolved the residue.

The *ethereal residue* on treating with cold ether was not entirely soluble in it. It was soluble in chloroform, benzole, and carbon di-sulphide; incompletely soluble in cold alcohol, and insoluble in amyl alcohol. The *ethereal residue* was treated with 95 per cent. alcohol, in which it was slightly soluble. A turbidity formed in the alcoholic solution on adding lead acetate, ferric chloride, ammonium hydrate, and sulphuric acid; it did not clear up on warming. Hydrochloric acid made a muddy mixture with the alcoholic solution. The *ethereal residue* was not entirely soluble in acetic ether; the latter separated coloring matter from it. The *ethereal residue*, insoluble in acetic ether and freed from coloring matter (chlorophyll), was a resinous substance. It melted at 80° C. The resin was boiled with absolute alcohol, and on throwing the alcoholic solution into cold water it was precipitated as a white cloud. It was not saponified.

Extract (4), *Yellow Base of Leaf.*

The residual powder from the petroleum spirit maceration was dried and extracted with stronger ether. The *ether extract* was a turbid yellow liquid; slightly acid in reaction. On evaporating the *ethereal extract* at the ordinary temperature a reddish-yellow granular solid remained. It melted at 79° C. For the determination of total solids extracted, a definite volume of the *extract* was evaporated, dried, and weighed.

I.

TOTAL SOLIDS.

Ethereal residue dried at 100° C	1.7 per cent of solids.
" " " 110° C	1.7 " " "
	-0.0 " " loss.

The *ethereal residue* was treated successively with distilled water, alcohol, and ether.

II.

Substances souble in ether and water	0.8 per cent.
" " " alcohol.	0.4 "
" insoluble in water and "	0.5 "
Total solids	1.7 "

The aqueous extract gave a neutral reaction with litmus. Negative results followed examination for tannin, gallic acid, glucosides, alkaloids, and any compounds containing nitrogen.

The *ethereal residue* (the *residue* insoluble in water) was an opaque reddish-yellow colored substance, and was identified as a resin. It melted at 79° C. It was insoluble in ether, benzole, chloroform, and acetic ether; incompletely soluble in cold absolute alcohol, amyl-alcohol, carbon di-sulphide, and oil of turpentine. It was soluble in aqueous and alcoholic soda. On boiling with them, it was saponified. Concentrated sulphuric acid dissolved the resin and colored it a yellowish-brown. Chloroform formed a turbid mixture with the acid solution. The action of strong nitric acid on the resin was slow. The resin was incompletely soluble in 95 per cent. alcohol. Lead acetate gave a cloudiness with the alcoholic solution which increased on boiling. Ferric chloride thickened the alcoholic solution, and on boiling it gave a yellow precipitate which was insoluble in acids, alkalies, absolute alcohol, and acetic ether. The chloroform extract gave no coloration with bromine solution.

SUMMARY II.

Ethereal Extracts.

	Solids extracted.	*Character of residue.*	*Reaction with litmus.*	*Melting Point.*	*Specific gravity*	*Substances soluble in ether and water.*	*Substances soluble in alcohol and water.*	*Substances soluble only in ether.*
1. The bark of the root.....	3.16 p. ct.	resin	acid	70° C.	1.091	traces	traces	.....
2. " wood "	1.70 "	"	slightly "	70° C.	1.091	"	"	.....
3. " green leaf.....	1.25 "	" chlorophyll	" "	80° C.	.....	0.34 p. ct.	0.15 p. ct.	0.65 p. ct.
4. " yellow base of leaf...	1.70 "	resin	" "	79° C.	.....	0.80 "	0.40 "	0.50 "

The residues from the *ether extracts* (1) and (2) of the bark and of the wood of the root contained resins which were identified as the same compound. They correspond in color, melting point, specific gravity, solubilities, and reactions. The resin is a transparent, ruby-colored substance, crystalline in structure, and of a softer consistency than ordinary resin. It was examined by Hirschsohn's scheme.* It differed from all described resins in its reactions with the reagents used to identify them. It is proposed to name it yuccal.†

Yuccal is imperfectly soluble in 95 per cent. alcohol; soluble in boiling absolute alcohol, in cold ether, and amyl alcohol; not appreciably soluble in chloroform, benzole, carbon di-sulphide, and alcoholic ammonia. Cold acetic ether dissolved the coloring matter from the resin, leaving a colorless solid. Hot acetic ether dissolved it perfectly. Yuccal when heated on platinum foil gave off as it burned a pleasant and aromatic

* Loc. cit.

† See foot-note, *ether extract* (2).

odor. Tests failed to show the presence of benzoic or cinnamic acids. A blood-red color reaction was obtained by warming yuccal with a crystal of ammonium molybdate and a few drops of strong nitric acid. Warm dilute nitric acid dissolved the resin, colorless; cold nitric acid gave a brownish-green color reaction. Yuccal was mixed with concentrated nitric acid and heated. After some time had passed, an energetic reaction occurred and nitrous fumes were given off. A yellowish-brown residue was one of the products of the reaction. This residue was almost insoluble in water, or acids. It was soluble in alcohol and potassium hydrate.*

As the *ether extracts* (1) and (2) were concentrated, white needle-shaped crystals appeared floating in the liquids, whose physical structure, and insolubility in water and acetic ether, suggested identical substances. The crystals separated from yuccal by the lead acetate method, already described, have not been sufficiently studied to identify them with the white needle-shaped crystals in the *ether extracts* nor with any class of chemical compounds. However, the absence of gallic acid, glucosides, and alkaloids in the aqueous extracts from the *ethereal residues*, would show that the crystals separated from yuccal are a constituent part of the resin.

The experiments with spirit of different strengths are only of value, so far as they were carried out, in showing the possibility of separating the resin into distinct parts.†

Tannin was not present in these *ethereal extracts.*

Ethereal extract (3) was green-colored and fluorescent from the chlorophyll of the leaves. On evaporating, the *ethereal residue* was amorphous and of a green color. The aqueous extract obtained from treating this *ethereal residue* was neutral in reaction and bitter to the taste. It contained neither gallic acid, tannin, nor glucosides. It was agitated with acetic ether, and the solvent removed a solid, which under the microscope proved to be white needle-shaped crystals arranged in bundles. Potash solution formed a yellow-colored mixture with the crystals, hydrochloric acid discharged the color. The subject has been too little studied to state definitely if these crystals are or are not identical with the crystals found in the *ethereal extracts* (1) and (2). But it should be noted, that unless the crystals from *Ethereal extract* (3) are brought into aqueous solution mechanically by some compound not present in *ethereal extracts* (1), and (2), the indications are in favor of the crystals from (3) not being identical with them; for the crystals from (1) and (2) were insoluble in water and not removed by acetic ether.

* See foot-note 3, *ether extract* (2).

† The amount of material on which these experiments, as well as others described in this paper, were tried, was too small in quantity for me to obtain more conclusive results. The facts which have been ascertained will serve as a guide in future investigations.

The *ethereal residue* was treated with acidulated water and tested negatively for alkaloids.

The *ethereal residue* insoluble in water was a mixture of two resins (I) and (II). The one (I) was dissolved by absolute alcohol, the other (II) was mostly soluble in ether. The alcoholic residue was crystalline. It was insoluble in acetic ether, but was saponified with alcoholic soda. The ether residue was a resin anhydride; it was insoluble in alcohol, and in alcoholic or aqueous alkalies.

The amorphous and green-colored *ethereal residue* was not entirely redissolved by cold ether. It was soluble in chloroform, benzole and carbon di-sulphide; incompletely soluble in cold alcohol, and insoluble in amyl alcohol. It was slightly soluble in 95 per cent. alcohol and in acetic ether. The latter separated the green coloring matter from it. The resinous mass insoluble in acetic ether melted at 80° C. It was not saponified. This resinous mass insoluble in acetic ether is a mixture of the two resins just described (I) and (II). It was noticeable that the resinous mass was not saponified. Resin (I) was saponified. Resin (II) did not saponify, and as this resin exceeded in amount by 0.5 per cent. resin (I), it would show that a certain percentage of resin anhydride in a mixture of two resins forbids the saponification of the mixture.

It was not determined if the crystals dissolved by water and separated by acetic ether were a part of resin (I) or resin (II) or an independent compound.

Ethereal extract (4) was a turbid yellow liquid. On evaporating, a reddish-yellow granular solid remained. The extract from the aqueous treatment was tested with negative results for tannin, gallic acid, glucosides, and alkaloids. The *ethereal residue* insoluble in water was identified as a resin. It was soluble in ether, benzole, chloroform, and acetic ether; incompletely soluble in cold absolute alcohol, amyl alcohol, carbon di-sulphide, and oil of turpentine. It was saponified. A resin was extracted by boiling absolute alcohol from the residual powder of the leaves (the yellow base) which was identified as the same resin, and the name of pyrophæal* was proposed for it.

I. Resins (1) and (2) are identical substances (yuccal).

II. *Ethereal residue* (3) is a mixture of two resins, and a crystalline principle soluble in water.

III. Resin (4) pyrophæal is identical with a resin found in *alcoholic extract* (4).

ALCOHOLIC EXTRACTS.

Extract (1), *Bark of the Root.*

The residual powder from the ether extraction was dried, and replaced in the percolator. The maceration was conducted at the boiling temperature of alcohol.

* Pyrophæal. Science, September 11, 1885.

Squibb's stronger alcohol was used. A dark red-colored liquid was extracted. It was neutral in reaction with litmus. The *alcoholic extract* was evaporated in a current of carbonic acid. The *residue* was non-crystalline and of a red color. A definite volume of the *alcoholic extract* was evaporated, dried until the weight remained constant, and the *residue* incinerated in a weighed platinum dish and the ash estimated.

I.

TOTAL SOLIDS.

Alcoholic residue dried at 100° C	9.25 per cent.
" " " 110° C	9.25 "
" " ash	0.2 "

The *alcoholic residue* was treated with distilled water, and a definite volume of the extract was evaporated, dried, and weighed. The *alcoholic residue* insoluble in water was treated with water containing ammonia (one part in fifty). This ammoniacal extract was evaporated with excess of acetic acid, and the residue rinsed with a little water on a filter, dried, and weighed. The dried aqueous extract insoluble in ammonia was then estimated.

II.

Distilled water residue	3.22 per cent.
Ammonia " "	5.43 "
Insoluble " "	0.60 "
Total solids	9.25 "

The aqueous extract from the *alcoholic residue* was studied as follows: It was not colored by a ferroso-ferric salt nor precipitated by gelatine and alum solutions, showing absence of gallic acid and tannin. A portion of the aqueous extract was acidified with sulphuric acid and agitated successively with petroleum spirit, benzole, chloroform, and amyl alcohol. The acidified liquid was rendered alkaline by ammonia and agitated with the solvents in the same order. Petroleum spirit removed from the acidified solution traces of an amorphous residue, soluble in sulphuric acid and caustic soda. Benzole and chloroform separated no substances from the solution. As the amyl alcohol solution was evaporating white needle-shaped crystals were seen floating in the liquid. On drying the residue they were decomposed and melted, leaving a dark-colored liquid. Several attempts were made to dry these crystals, without success. A few of the crystals were recovered from the solution, and tested for alkaloids; no reactions were obtained with the usual reagents for them.

Glucose was estimated from the aqueous extract. The liquid was heated over a water bath with Fehling's solution, and the precipitated red cuprous oxide was thrown upon a weighed filter, dried, and incinerated. The glucose was estimated gravimetri-

cally by calculating the amount of cupric oxide. It yielded 0.619 per cent. A portion of the aqueous extract was boiled with acid, neutralized, and heated over a water bath with Fehling's solution to calculate, by difference, saccharose or other reducible compounds, and by this method 0.18 per cent. was obtained.

The *alcoholic extract* was described as being deeply colored. This coloring principle* was completely precipitated by sub-acetate of lead. The lead precipitate was collected on a filter, suspended in water, and decomposed by sulphuretted hydrogen, filtered, and the filtrate freed from all odor. It was allowed to evaporate slowly over sulphuric acid. The residue was a brownish-gray mass, interspersed with fine crystals which radiated from a nucleus. The mass was weighed and gave 3.27 per cent. of solids. Another portion of the *alcoholic extract* was agitated with water and acetic ether. The coloring matter was taken up by the acetic ether, and on evaporating a red-colored substance was recovered. It was dried and weighed, yielding 2.2 per cent. This red-colored residue was perfectly soluble in cold water. This solution was tested with the following reagents: It gave with potassium bichromate a creamy-colored precipitate; ferric chloride, a yellowish-green precipitate; ferrous sulphate a reddish-brown precipitate; stannous chloride, no precipitate, a yellow cloudy liquid; alum, a cloudy solution; neutral acetate of lead, a slight precipitate. The red color of the coloring matter was brought out on addition of alkalies. It was destroyed by acids.

Extract (2), *Wood of the Root.*

The residual powder from the ether treatment was dried and macerated with Squibb's stronger alcohol. The *alcoholic extract* was neutral in reaction; when warm it was a clear reddish-golden-colored liquid. On cooling, a creamy-white solid settled at the bottom of the flask. This substance was soluble in water, and was identified as saponin by the usual tests for it. A definite volume of the *alcoholic extract* was evaporated in a current of carbonic acid, dried, and weighed. The residue was incinerated in a weighed platinum crucible for the ash determination.

TOTAL SOLIDS.

Alcoholic residue dried at 100° C	14.3 per cent.
" " " 110° C	14.3 "
" " ash	00.1 "

The *alcoholic residue* was treated with cold water in which it was soluble. A cloudy solution was formed, and on shaking, it became frothy, and presented the appearance of an emulsion. It was allowed to stand for several days to see if the resinous matter separated, but the emulsion was permanent, as no separation had

* A red crystalline coloring matter. Science, September 11, 1885.

taken place. The emulsified liquid was agitated with acetic ether, and this solvent readily separated most of the resin from the aqueous portion. The water extract was then evaporated to dryness and redissolved in water. Gelatine and alum solution did not precipitate the extract, showing absence of tannin; no coloration with iron salts, absence of gallic acid; negative results followed tests for alkaloids; the aqueous extract was boiled with potash and no ammonia fumes were formed; adding gold chloride, and potassio-mercuric iodide solutions to the extract gave no precipitate. A measured portion of the aqueous extract was acidified with sulphuric acid, and agitated successively with petroleum spirit, benzole, and chloroform. The solvents were evaporated; petroleum spirit removed 0.01 per cent of a resinous substance, imperfectly soluble in cold and boiling aqueous alkalies, dissolved by sulphuric acid with a red coloration; chloroform left a brownish residue which, on weighing, yielded 0.4 per cent. This residue was moistened with a few drops of concentrated sulphuric acid, and changed to a red-violet color characteristic of saponin.

A certain portion of the aqueous extract was rendered alkaline, and heated over a water bath with Fehling's solution. The precipitated copper was collected on a weighed filter, dried, and incinerated, and the glucose estimated gravimetrically from it. It yielded 1.592 per cent. Another portion of the aqueous extract was acidified, boiled, and potash added until the solution was alkaline to litmus paper, then the liquid was mixed with Fehling's solution and heated over a water bath. The percentage of saccharose or other substances which reduced the copper was calculated by difference. It amounted to 0.929 per cent.

The resin separated by acetic ether was an opaque substance, greenish-yellow in color, and insoluble in ether. The resin was dissolved in water and frothed on shaking. The emulsion in this case was not quite so permanent, as a slight resinous sediment settled after a time, possibly due to changes in the resin through oxidation.

Extract (3), *the Green Part of the Leaf.*

The dried residual powder was macerated by the aid of heat with Squibb's stronger alcohol. When warm the *alcoholic extract* was clear, but on cooling the solution became cloudy, and a creamy-white fine precipitate settled. The *alcoholic extract* was neutral in reaction. It was evaporated in a current of carbonic acid, dried, and weighed. A certain part of the residue was incinerated and the ash determined.

TOTAL SOLIDS.

Alcoholic residue dried at 100° C.	3.80 per cent.
" " " 110° C.	3.80 "
" " ash	0.15 "

The *alcoholic residue* was treated with cold distilled water. It had a slightly acid reaction with litmus. An emulsion was formed on the addition of water to the *alcoholic residue.* A measured quantity of it was evaporated, dried, and weighed. It amounted to 3.4 per cent., 0.4 per cent. of the *alcoholic residue* was insoluble in water. Tannin, gallic acid, and alkaloids were tested for and with negative results.

The liquid from the aqueous treatment of the *alcoholic residue* was rendered alkaline, and boiled with Fehling's solution, and there was no reduction. Boettger's bismuth test was also tried and with negative results. The aqueous portion was boiled with acid and examined in the usual way for glucosides; the results were negative.

One volume of the aqueous solution was mixed with three volumes of stronger alcohol. It was placed on ice, and after some time a white precipitate formed. The precipitate was collected and dissolved in water. It frothed on shaking. On addition of a concentrated solution of caustic baryta, a creamy-white precipitate of saponin-baryta was obtained. Sulphuric acid gave the usual red-violet color reaction with the precipitate from the alcoholic aqueous solution.

The method of successive agitation of an aqueous extract with solvents already described was followed. Petroleum spirit on evaporating left a resinous substance. The residue separated by chloroform from an acidified solution was a brownish-colored substance. It was soluble in water, and frothed on shaking. It was colored red-violet by sulphuric acid, and the aqueous solution was precipitated by barium hydrate. Chloroform separated a brownish solid from an alkaline aqueous solution. It was precipitated by barium, colored red-violet by sulphuric acid, and its aqueous solution frothed on shaking. This brownish residue was identified as saponin.

Extract (4), *the Yellow Base of the Leaf.*

The residual powder, dried from all traces of ether, was macerated with hot alcohol. The *alcoholic extract* was a currant-colored liquid, and slightly acid in reaction. The liquid became clear on standing, and a creamy-white solid, identified as saponin, separated from it. The *alcoholic extract* was evaporated, dried, and weighed, and the ash of the residue was estimated.

TOTAL SOLIDS.

Alcoholic residue dried at 100° C.	4.30 per cent.
" " " 110° C.	4.30 "
" " ash.	0.05 "

The *alcoholic residue* was treated with cold distilled water. The solution was slightly colored, and faintly acid in reaction. The absence of gallic acid, tannin, and alkaloids, was determined by negative results with iron salts, gelatine and alum solu-

tion, gold chloride, and potassio-mercuric solutions. Acetate of lead caused no precipitation. Fehling's solution detected a trace of glucose.

An imperfect emulsion formed on adding water to the *alcoholic residue*. Upon standing, the resin settled; the liquid was filtered several times, and the greater part of the resin collected. It was an opaque reddish-yellow-colored substance. It had the same melting point (79° C.), solubilities, and physical appearance as the resin of *ether extract* (4). The resin was examined by Hirschsohn's scheme. It differed in character from the many resins described by that author, and it is proposed to name it pyrophæal.*

Pyrophæal was slightly soluble in ether, and 95 per cent. alcohol; soluble in benzole, chloroform, and acetic ether; incompletely soluble in cold absolute alcohol, amyl alcohol, carbon di-sulphide, and oil of turpentine. It was saponified with aqueous and alcoholic soda. The ethereal resin solution was cloudy. The alcoholic resin solution gave a precipitate with lead acetate which did not disappear on boiling; ferric chloride and aqueous ammonia formed turbid mixtures with it. The chloroform resin solution was not affected by bromine solution. The petroleum-ether-resin solution turned to a turbid mixture on adding iodine solution. Alcohol containing hydrochloric acid was not colored by the resin. Sulphuric acid and alcohol gave a turbid brown mixture with it, and sodium carbonate solution was colored pale brown when cold or on warming.

SUMMARY III.

Alcoholic Extracts.

	Solids extracted.	*Character of residue.*	*Reaction with litmus.*	*Quantitative estimation of glucose.*	*Quantitative estimation of saccharose or other reducible compounds.*
1. The bark of the root.....	9.25 p. ct. 0.20 p. ct. ash	red coloring matter crystalline	neutral	0.619 p. ct.	0.180 p. ct.
2. " wood "	14.30 " 0.10 "	resin, saponin	"	1.592 "	0.929 "
3. " green leaf.....	3.80 " 0.15 "	resins, "	"	none	none
4. " yellow base of leaf.....	4.30 " 0.05 "	resin, "	slightly acid	traces	traces

Extract (1).

My attention was not directed to the presence of saponin in *extract* (1), for the characteristic properties which it imparted to *extracts* (2), (3), and (4), were absent; but, it was evident that saponin was present in the bark, for on boiling the latter in distilled water, the presence of the compound was indicated. The solution frothed on shaking, and by adding a concentrated solution of caustic baryta, saponin-baryta was precipitated.†

* Pyrophæal. Science, September 11, 1885.

† Saponin in the bark of *Yucca angustifolia*. Science, September 11, 1885.

A coloring matter* contained in the bark was extracted, and imparted to the *alcoholic extract* a brilliant red color. It was precipitated by sub-acetate of lead, and the lead precipitate suspended in water and decomposed by sulphuretted hydrogen. The lead sulphide filtrate was evaporated over a water bath until the odor of sulphuretted hydrogen was expelled, and the concentrated liquid was placed over sulphuric acid to evaporate slowly. A crystalline residue was obtained. On addition of alkalies to the colorless lead sulphide filtrate the red color of the original solution was developed. Acid discharged the color. Acetic ether took up the red colored substance. The acetic ether residue was a red uniform solid, and soluble in water. It was precipitated from the aqueous solution by sub-acetate of lead, potassium bichromate, ferric chloride, ferrous sulphate, and it was clouded by alum, and stannous chloride solutions.

Tannin, gallic acid, and alkaloids were absent.

Amyl alcohol separated from the acidified aqueous extract white needle-shaped crystals. It was not determined if these crystals were the same as those of the coloring matter.

Extracts (2), (3), *and* (4).

Extracts (2) and (3) when warm were clear, and on cooling a creamy-white solid separated. Extract (4), if warmed, was turbid, and as the liquid cooled, a creamy-white substance remained at the bottom of the flask, and the supernatant fluid became clear. This creamy-white substance was identified in each of the *extracts* as saponin.†

The results following an aqueous treatment of *alcoholic residues* (2) and (3), were noticeable. The *residues* were dissolved, and by shaking the mixtures, emulsified. This emulsion was permanent, as no resinous matter separated on standing several days. The emulsion was agitated with acetic ether, and by this means, most of the resin and saponin were separated from the aqueous portion. The saponin was removed mechanically with the resin, as it is almost insoluble in acetic ether. The resin-saponin mass was insoluble in ether, soluble in water. The solution frothed on shaking and emulsified, but the emulsion was not so permanent as in the first case, for a resinous sediment settled after a time. Chloroform separated saponin from an acidified aqueous solution, and also from an alkaline aqueous solution of the *residues;* and the red-violet saponin reaction with concentrated sulphuric acid was obtained.

The solubility in water of the *alcoholic residues* (2), (3), and (4), and the resulting emulsion were unusual, and explicable by the facts collected from a series of ex-

* A red crystalline coloring matter. Science, September 11, 1885.

† Saponin in the wood of the root and leaves. Science, September 11, 1885.

periments with resins and saponin, since I had successfully emulsified resins with aqueous and alcoholic saponin solutions.*

By hot alcoholic treatment the *yucca* yielded a residue of saponin and resin which became emulsified on the addition of water, giving results identical with those of the resins above described.†

Extract (4) contained a resin. It was an opaque reddish-yellow colored substance, and it differed, by its reactions, from the many resin classes given in Hirschsohn's scheme. It is proposed to name it pyrophæal.‡ A resin having the same melting point, solubilities, physical appearance, and chemical reactions, was discovered in the *ethereal extract* (4). It was identified as the same compound for which the name pyrophæal is proposed.

Tannin, gallic acid, and alkaloids were not detected in *extracts* (2), (3), and (4). In *extract* (3) glucose was not found.

The Solids of the Alcoholic Extracts.

I. A red coloring matter (crystalline).
II. A new resin (yuccal).§
III. A second new resin (pyrophæal).§
IV. A mixture of a crystalline resin and a resin anhydride.
V. Saponin.§
VI. Glucose, and saccharose or other reducible compounds.
VII. Ash.

AQUEOUS EXTRACTS.

Extract (1), *Bark of the Root.*

The residual powder was thoroughly dried from alcohol. It was returned to the percolator, and cold distilled water added until a definite amount had been used. The *aqueous extract* was dark colored, and of a faintly acid reaction. A certain quantity of the *extract* was evaporated, dried, and weighed. From a known weight of the *aqueous residue*, the ash was calculated. The incineration was conduced in a covered porcelain crucible of known weight.

*The same kinds of resins were used in these experiments as in those with which I determined the solubility of resins in acetic ether. See foot-note (2), *ethereal extract* (2).

† It was not until a later date following the time of these experiments that I found a reference to saponin-resin emulsion in L'Officine ou Répertoire General de Pharmacie Pratique, par Dorvault. Huitième Edition, Paris, 1872, p. 816. Also, refer to examination of the *Yucca angustifolia*, by H. C. De S. Abbott, published in the Medical and Surgical Reporter, Philadelphia, September 12, 1885, page 301.

‡ Pyrophæal, loc. cit.

§ Science, September 11, 1885, page 210. Abstract of a paper on the chemical study of *Yucca angustifolia*, by H. C. De S. Abbott.

TOTAL SOLIDS.

Aqueous residue dried between 100° C and 110° C.	4.00 per cent.
" " ash.	2.65 "

Gum.

One volume of the *aqueous extract* was mixed with two volumes of Squibb's stronger alcohol. The mixture was kept in a cool place for twenty-four hours, and the precipitate which had formed was collected on a weighed filter, washed with 66 per cent. alcohol, dried, and weighed. The precipitate and filter were incinerated in a weighed porcelain crucible, and the weight of the filter being deducted, the percentage of ash was determined.

Weight of precipitate by stronger alcohol yielded	2.0 per cent.
" " ash yielded	0.2 "

Another portion of the *aqueous extract* was precipitated by stronger alcohol, and the precipitate consisted of gum and albuminous substances. It was incompletely soluble in water. The soluble matter was gum, it was recovered from solution by evaporating the liquid to dryness. The gummy residue was almost completely soluble in cold water. It was precipitated from a concentrated aqueous solution by stronger alcohol; basic acetate of lead precipitated it as a flocculent precipitate. Borax did not thicken the gum solution, and ferric chloride and sodium chloride solutions did not precipitate it. The gum was boiled with dilute acid, and heated over a water bath with Fehling's solution which it reduced. A few drops of hydrochloric acid and stronger alcohol were mixed with the concentrated gum solution for the separation of arabin. It was not separated.

Carbhydrates.

The filtrate and wash alcohol from the gum precipitate were mixed, and evaporated to a syrupy consistency at a temperature of 70° to 80° C. The concentrated solution was treated with four volumes of stronger alcohol, and the resulting precipitate of *carbhydrates* rapidly filtered off. It was soluble in water. It was not precipitated from aqueous solution by basic acetate of lead, and by this means it was distinguished from vegetable mucilage. The *carbhydrates* were boiled with dilute acid, and the solution was rendered alkaline, and heated over a water bath with Fehling's solution. The latter was reduced. The percentage of *carbhydrates* as estimated, amounted to 0.2 per cent. An aqueous *carbhydrate* solution was mixed with a solution of barium in 40 per cent. alcohol. It yielded no precipitate.

Carbhydrate Filtrate.

The *carbhydrate filtrate* was concentrated at a low temperature in a current of carbonic acid until the alcohol was dissipated. The *residue* was examined for glucose, organic acids, saponin, and tannin. Traces of glucose were detected qualitatively by Fehling's test; the amount of cuprous oxide present was too small to estimate gravimetrically. A part of the *carbhydrate filtrate residue* was boiled with 83 per cent. alcohol, and filtered while hot. On cooling, a precipitate formed. This precipitate was identified as saponin. It was almost insoluble in stronger alcohol. Baryta-water precipitated it from aqueous solution. Its aqueous solutions frothed on shaking. When agitated with chloroform and on evaporating the chloroform solution, a light-colored residue was obtained. A few drops of concentrated sulphuric acid mixed with it gave a reddish-violet color reaction. Another portion of the *carbhydrate filtrate residue* was precipitated with neutral acetate of lead and filtered. The precipitate was suspended in water, decomposed by sulphuretted hydrogen, and the lead sulphide filtrate evaporated over a water-bath to expel all odor of sulphuretted hydrogen. The liquid was cooled and lime-water added until the reaction was alkaline to litmus. A turbidity formed when the lime-water was added to the filtrate, and was not entirely cleared on the addition of dilute acetic acid. A neutralized portion of the lead sulphide filtrate gave a yellow precipitate with a ferrous salt. Oxalic acid by these tests was indicated, and possibly other vegetable acids were present in the filtrate. The *carbhydrate filtrate residue* was examined for tannin, and with a negative result. Calcium oxalate was separated.

Extract (2), *the Wood of the Root.*

The powder used in the alcoholic maceration was thoroughly dried, and replaced in the percolator. A measured quantity of cold distilled water was allowed to percolate slowly through the powder. The *extract* was colored, and slightly acid in reaction. A definite volume of the *extract* was evaporated, dried, and weighed. A known weight of the *residue* was incinerated in a weighed covered porcelain crucible, and the ash determined. The ash was white and incompletely soluble in water.

TOTAL SOLIDS.

Aqueous residue dried between 100° and 110° C.	12.10 per cent.
" " ash	1.74 "

Gum.

A certain quantity of the *aqueous extract* was mixed with two volumes of stronger alcohol (Squibb's). The mixture was allowed to stand for twenty-four hours, and the

precipitate which formed was collected on a weighed filter. It was dried, and weighed. The precipitate and filter were incinerated in a weighed porcelain covered crucible, and the percentage of ash calculated.

Weight of precipitate by stronger alcohol yielded	1.70 per cent.
" " ash yielded	0.34 "

Carbhydrates.

The filtrate and wash-alcohol from the gum precipitate were concentrated at a low temperature, and the residue was mixed with four volumes of stronger alcohol. The precipitate was rapidly filtered off, and the percentage of *carbhydrates* calculated gravimetrically in the usual way, from the amount of cupric oxide reduced from Fehling's solution. It yielded 2.75 per cent.

Carbhydrate Filtrate.

A portion, representing a certain volume of the *aqueous extract*, of the *carbhydrate filtrate* was evaporated, dried, and weighed. It yielded 7.65 per cent. of the total solids of the *aqueous extract residue.* A known weight of the *carbhydrate filtrate residue* was dissolved in water and heated over a water-bath with Fehling's solution, and the amount of glucose present estimated gravimetrically from the weight of the cupric oxide. It was estimated as 4.47 per cent. Another portion of the *carbhydrate filtrate residue* was boiled with 83 per cent. alcohol. A precipitate formed on cooling, which was collected on a weighed filter, dried, and weighed. It yielded 1.98 per cent. The 83 per cent. alcohol precipitate was identified as saponin by the usual tests. The *carbhydrate filtrate residue* was precipitated by acetate of lead and the precipitate examined qualitatively for organic acids. The lead precipitate was decomposed by sulphuretted hydrogen and filtered, and the filtrate concentrated over a water-bath, and mixed with lime-water until turbid. The turbidity did not clear on adding dilute acetic acid.

The *aqueous extract* was examined for tannin, and with negative results. Calcium oxalate was present.

Extract (3), *the Green Part of the Leaf.*

The residual powder was dried from all traces of alcohol, and cold distilled water was allowed to slowly percolate through the powder. The *extract* was colored, and slightly acid in reaction. A definite volume of the *extract* was evaporated, dried, and weighed, and the ash calculated from incinerating a known weight of the *residue.*

TOTAL SOLIDS.

Aqueous residue dried between 100° C and 110°	4.35 per cent.
" " ash	0.40 "

Gum.

One volume of the *aqueous extract* was mixed with two volumes of stronger alcohol. The precipitate was collected after twenty-four hours, washed with 66 per cent. alcohol, dried, and weighed. The precipitate and filter were incinerated, and the ash estimated.

Weight of precipitate by stronger alcohol yielded	0.775 per cent.
" " ash yielded	0.125 "

Carbhydrates.

The gum filtrate and wash alcohol were concentrated at a low temperature, and the residual liquid mixed with four volumes of stronger alcohol. The resulting precipitate was rapidly filtered and collected. The percentage of *carbhydrates* was estimated gravimetrically from a copper solution in the usual way. It amounted to 0.525 per cent.

Carbhydrate Filtrate.

The *filtrate* was evaporated to dryness. A part of the *residue* was boiled with 83 per cent. alcohol. On cooling, a precipitate formed. It was identified by the usual tests as saponin. Another portion of the *residue* was precipitated with basic acetate of lead. The lead precipitate was decomposed by sulphuretted hydrogen, the solution filtered, and the filtrate evaporated over a water bath until all odor of sulphuretted hydrogen was dissipated. A part of the lead sulphide filtrate was mixed with lime-water, and a precipitate formed not completely dissolved by acetic acid. The remainder of the lead sulphide filtrate was allowed to evaporate over sulphuric acid. The residue consisted of a mass of fine crystals radiating from a centre.*

The crystals gave a very acid reaction when placed on moistened blue litmus paper. They turned black and left a residue when heated on platinum foil, and the residue was slowly dissolved by nitric or hydrochloric acid. The quantity of residue was very small, and no effervescence was observed. The melting point of the crystals was taken. A small quantity was placed in a tube with thin walls, and gradually heated; at 150° C. the substance sublimed, leaving a white, cloudy stain on the inner surface of the tube; at 190° C., this cloudy stain changed to a pale green spot, and with increasing temperature to 210° C., no further change was noted. Dry sodium carbonate was added to an aqueous solution of the crystals, and a slight effervescence was observed. Some iron was separated which possibly was in combination with the

* The material used in this analysis of the green part of the leaf was quite dry and powdered readily. A previous examination of the *fresh leaves* gave more satisfactory quantitative results. A gum was extracted which promises to be of interest for a future study, and the crystals separated from the lead sulphide filtrate are to be further investigated.

crystalline principle. Negative results followed tests for formates, acetates, malates, tartrates, citrates, phosphates, oxalates, alkaloids, and glucosides.

The amount of glucose present in the *aqueous extract* was too small to determine quantitatively. Negative results for tannin.

Extract (4), *the Yellow Base of the Leaf.*

The residual powder from the alcoholic maceration was dried and replaced in the percolator. Cold distilled water was allowed to percolate slowly through the powder. The *aqueous extract* was slightly acid in reaction. A known measure of it was evaporated, dried, and weighed. The ash was determined from a part of the *aqueous extract residue.*

TOTAL SOLIDS.

Aqueous residue dried between 100° and 110° C	11.35 per cent.
" " ash	3.10 "

Gum.

One volume of the *aqueous extract* was mixed with two volumes of stronger alcohol. The precipitate was collected, washed with 66 per cent. alcohol, dried, and weighed. The ash was calculated from incinerating the precipitate, and deducting the filter.

Weight of precipitate by stronger alcohol yielded	3.850 per cent.
" " ash yielded	0.676 "

Carbhydrates.

The filtrate and wash alcohol from the gum precipitate were concentrated at a low temperature. The residual liquid was mixed with four volumes of stronger alcohol, when a precipitate formed, and was rapidly filtered off. The *carbhydrates* were dissolved in water, boiled with dilute acid, and the liquid rendered alkaline and heated over a water bath with Fehling's solution. The amount of carbhydrates was estimated gravimetrically in the usual way. It gave 2.95 per cent.

Carbhydrate Filtrate.

The *filtrate residue* was examined for glucose, and traces of it were present. The *filtrate residue* was precipitated with acetate of lead, and the lead precipitate was dissolved in water and decomposed by sulphuretted hydrogen. The lead sulphide filtrate was tested qualitatively for organic acids, and a turbidity formed on adding to the filtrate lime-water. It was not completely cleared by acetic acid.

Negative results followed tests with alcoholic methyl-violet solution for mineral acids. The *aqueous extract* contained no tannin. Calcium oxalate was determined in it.

AQUEOUS MACERATION AT A TEMPERATURE OF 50° C. TO 60° C.

The Bark of the Root (1), *the Wood of the Root* (2).

The powder (1) used in the cold water extraction was macerated with distilled water heated between 50° and 60° C. The *warm aqueous extract* (1) was cooled and mixed with three volumes of stronger alcohol. A precipitate formed, it was dried, weighed, and the percentage estimated. It yielded 0.03 per cent.* The precipitate was dissolved in warm water. On evaporating the filtrate, a white residue was obtained. It was stained yellow by iodine.

The powder (2) from the cold water treatment was macerated in the warm water. The *warm aqueous extract* (2) was a dark colored liquid, indicating a coloring matter. A certain measure of the *extract* was evaporated, and the solids estimated. It amounted to 4 per cent. The percentage of solids precipitated from the *extract* by stronger alcohol was 0.25 per cent.

QUANTITATIVE ESTIMATION OF SAPONIN.†

The two methods of Christophsohn and Otten for the quantitative estimation of saponin were adopted. The wood of the root was examined.

A.—Ten grams of the original powder were boiled with distilled water. The saponin was precipitated by baryta-water. After weighing, it was ignited, and the baryta estimated as carbonate, calculated into oxide and deducted from the weight of the saponin-baryta, the difference being the weight of saponin.

B.—The saponin-baryta was decomposed by acid and the weight of the sapogenin was ascertained and calculated to saponin.

Several estimations were made on two specimens of the Yucca, collected at different times of the year.

Mean percentage, A ..... 8.95 per cent.
" " B ..... 10.40‡ "

SUMMARY IV.

Aqueous Extracts.

	Solids extracted	*Ash.*	*Gum.*	*Ash.*	*Glucose.*	*Saponin.*	
						A.	*B.*
1. The bark of the root.....	4.00 p. ct.	2.65 p. ct.	2.00 p. ct.	0.20 p. ct.	traces	.....	.....
2. " wood "	12.10 "	1.74 "	1.70 "	0.34 "	4.47 p. ct.	8.95 p. ct.	10.40 p. ct.
3. " green leaf.....	4.35 "	0.40 "	0.77 "	0.12 "	traces	.....	.....
4. " yellow base of the leaf..	11.35 "	3.10 "	3.85 "	0.67 "	"	.....	.....

* *Examination for Inulin*, page 87, Plant Analysis, G. Dragendorff. English translation.

† Loc. cit., page 68.

‡ Examination of the *Yucca angustifolia*, by H. C. De S. Abbott. The Medical and Surgical Reporter, Sept. 12, 1885, p. 301.

The *aqueous extracts* contained gum, albuminous substances, carbhydrates, glucose, saponin, organic acids, calcium oxalate, and no tannin, mineral acids, nor alkaloids. Arabin was not separated from gum (1). Calcium oxalate was brought into aqueous solution possibly by means of the organic acids or saponin. Needle-shaped crystals were found in *extract* (3). They did not respond to tests for formates, acetates, malates, citrates, tartrates, phosphates, oxalates, glucosides, and alkaloids.

Aqueous extracts of 50° C. to 60 °C. from the bark and wood of the root contained Inulin.

DILUTE CAUSTIC SODA EXTRACTS.

Extract (1), *the Bark of the Root.*

The residual powder insoluble in water was suspended whilst moist in a dilute soda solution (0.1 to 0.2 per cent). After twenty-four hours the mixture was filtered. One volume of the filtrate was acidified with acetic acid and mixed with three volumes of 90 per cent. alcohol, and allowed to stand in the cool. The precipitate was collected, washed with 75 per cent. alcohol, dried, and weighed, deducting ash. It consisted of mucilaginous substances and albuminoids.

Weight of precipitate by 90 per cent. alcohol yielded	0.85 per cent.
" " ash yielded	0.25 "

Lassaigne's test showed the presence of albuminous substances.

The filtrate and wash alcohol from the 90 per cent. alcohol precipitate was evaporated to dryness, and weighed, deducting the amount of soda acetate. It gave 0.24 per cent. The residue soluble in water was mixed with acetate of copper solution. A very small quantity of albuminous substances was precipitated by the reagent.

The albuminoids of the bark were estimated from the total nitrogen in one gram of the original powder. It yielded 4.75 per cent of albuminoids.

The powder insoluble in dilute soda solution was washed with distilled water. The liquid was deeply colored. It was evaporated, and the amount of solids estimated. It gave 1.3 per cent.

Extract (2), *the Wood of the Root.*

The powder insoluble in water was treated in the same way as in *extract* (1). The filtered solution was mixed with 90 per cent. alcohol in the manner described. The precipitate was estimated, deducting ash.

Weight of precipitate by 90 per cent. alcohol yielded	2.170 per cent.
" " ash yielded	0.256 "

The filtrate from the 90 per cent. alcohol precipitate was treated with water, and the soluble matter precipitated by copper acetate. The precipitate was collected, dried,

weighed, and ignited, the resulting oxide of copper being deducted. It yielded 0.104 per cent. of albuminoids. A current of washed carbonic acid was passed through the *dilute soda extract* to determine the presence of globulin (vitellin, myosin), and with negative results. The albuminoids were determined from the total nitrogen in the powdered wood. It amounted to 4.75 per cent. The total albuminoids in the leaves gave 9.62 per cent.

DILUTE HYDROCHLORIC ACID EXTRACTS.

Extract (1), *the Bark of the Root.*

The powder insoluble in dilute soda was washed with water and suspended in water containing 1 per cent. of hydrochloric acid. The absence of the blue color which the starch granules assume when treated with iodine solution was determined by examining the bark under the microscope; and consequently it was not looked for in the *extract*. A qualitative test showed the presence of calcium phosphate, and calcium oxalate. A measured quantity of the *filtrate* was neutralized with ammonia and mixed with three volumes of 90 per cent. alcohol. The precipitate was collected on a weighed filter, washed with 60 per cent. alcohol, dried, and weighed. It was incinerated, and the ash deducted from the precipitate.

The precipitate yielded	5.20 per cent.
" " " of ash	0.98 "
Organic substance	4.22 "

The filtrate from the 90 per cent. alcohol precipitate was evaporated. The residue was composed of ammonium chloride, from the reagents employed, and an organic substance having an odor like gum benzoin. It was agitated with ether and on evaporating the solvent a white residue with an odor like benzoin was obtained. Sulphuric acid gave a red color with it. The amount of this substance was calculated. It gave 0.45 per cent.

Extract (2), *the Wood of the Root.*

The insoluble powder from the dilute soda maceration was washed with distilled water, and suspended in water containing 1 per cent. of hydrochloric acid. The same means were used as in *extract* (1) to determine the absence of starch in the wood of the root. Parabin was also absent. Calcium oxalate was detected by qualitative tests. A similar method, as employed for its estimation in extract (1), was used to determine it quantitatively.

The precipitate yielded	0.305 per cent.
" " " of ash	0.155 "
Organic substance	0.150 "

Extract (3), *the Green Part of Leaf.*

The powder used in the dilute soda maceration was washed with distilled water and suspended in water containing 1 per cent. of hydrochloric acid. Iron and calcium phosphate were detected in the extract. The leaves were examined under the microscope, and a blue color was developed by an aqueous solution of iodine, indicative of starch granules. Starch was also present in the yellow base of the leaves.

TOTAL QUANTITATIVE RESULTS.

	(1.) *The bark of the root.*	(2.) *The wood of the root.*	(3.) *The green part of leaf.*	(4.) *The yellow base of leaf.*
1. Moisture	06.78 per cent.	11.67 per cent.	08.11 per cent.	37.00 per cent.
2. Total ash	17.38 "	15.75 "	05.75 "	10.63 "
3. Petroleum spirit extract	01.24 "	00.55 "	02.20 "	01.10 "
4. Ethereal extract	03.16 "	01.70 "	01.25 "	01.70 "
5. Alcoholic "	09.25 "	14.30 "	03.80 "	04.30 "
6. Aqueous "	04.03 "	16.10 "	04.35 "	11.35 "
7. Dilute soda "	01.09 "	02.41 "	.....	.....
8. Wash residue	01.30 "	.....	.....	.....
9. Dilute acid extract	05.65 "	00.30 "	.....	.....
Total percentage	49.88 "	62.78 "	25.46 per cent.	66.08 per cent.
Total albuminoids estimated on powder.	04.75 "	04.74 "	09.62 "	.....
Quantitative saponin determination on powder by *A* and *B*	.....	*A.* 08.95 per cent.	.....	.....
	.....	*B.* 10.40 "	.....	.....

In my paper on The Chemical Study of *Yucca angustifolia*, read at Ann Arbor, Mich., I stated what methods I had employed to separate saponin, and the properties of the compound as observed in that plant. Since which time a further study of it has induced me to withhold the notes used at Ann Arbor, from this, and to offer them, with those collected later, in a separate and more complete publication.

I am indebted to Dr. F. M. Endlich, for his courteous consideration and kindness in selecting and forwarding the fine specimens of *Yucca* which were used in these analyses, and which were grown in the neighborhood of Lake Valley, New Mexico. Within a few weeks I have received, in addition, several hundred pounds of the plant from Dr. Endlich.

The investigations described in the preceding pages were conducted in the chemical laboratory of the Philadelphia College of Pharmacy, from February to August, 1885.